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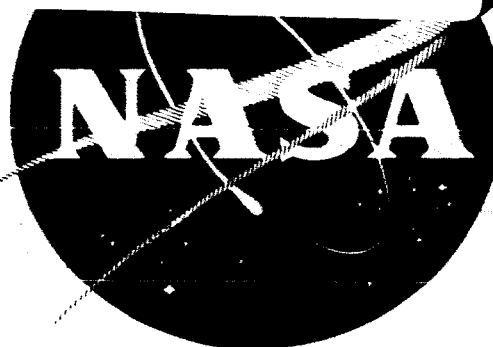
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## ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

Quarterly Progress Report No. 5  
For Quarterly Ending July 15, 1966

By  
R. W. HARRISON  
and  
E. E. HOFFMAN

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MISSILE AND SPACE DIVISION

GENERAL  ELECTRIC

CINCINNATI, OHIO 45215

ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

QUARTERLY PROGRESS REPORT 5

Covering the Period  
April 15, 1966 to July 15, 1966

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Prepared for  
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Under Contract NAS 3-6474

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NASA - Lewis Research Center  
Space Power Systems Division  
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SPACE POWER AND PROPULSION SECTION  
MISSILE AND SPACE DIVISION  
GENERAL ELECTRIC COMPANY  
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## FOREWORD

The work described herein is sponsored by the National Aeronautics and Space Administration under Contract NAS 3-6474. For this program, Mr. R. L. Davies is the NASA Project Manager.

The program is being administered for the General Electric Company by Dr. J. W. Semmel, Jr., and E. E. Hoffman, is acting as the Program Manager. J. Holowach, the Project Engineer, is responsible for the loop design, facilities, procurement and test operations. R. W. Harrison, the Project Metallurgist, is responsible for the materials procurement, utilization and evaluation aspects of the program. Personnel making major contributions to the program during the current reporting period include:

Alkali Metal Purification and Handling - Dr. R. B. Hand, L. E. Dotson, H. Bradley, and J. R. Reeves.

Welding and Joining - W. R. Young.

Refractory Alloy Procurement - R. G. Frank and L. B. Engel.

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## ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

### I. INTRODUCTION

This report covers the period from April 15, 1966 to July 15, 1966, of a program to fabricate, operate for 10,000 hours, and evaluate a potassium corrosion test loop constructed of T-111 (Ta-8W-2Hf) alloy. Materials for evaluation in the turbine simulator include Mo-TZC and Cb-132M. The loop design will be similar to the Prototype Loop; a two-phase, forced convection, potassium corrosion test loop which has been developed under Contract NAS 3-2547. Lithium will be heated by direct resistance in a primary loop. Heat rejection for condensation in the secondary loop will be accomplished by radiation in a high vacuum environment to the water cooled chamber. The compatibility of the selected materials will be evaluated at conditions representative of space electric power system operating conditions, namely:

- a. Boiling temperature, 2050°F
- b. Superheat temperature, 2150°F
- c. Condensing temperature, 1400°F
- d. Subcooling temperature, 1000°F
- e. Mass flow rate, 40 lb/hr
- f. Boiler exit vapor velocity, 50 ft/sec
- g. Average heat flux in plug (0-18 inches), 240,000 BTU/hr ft<sup>2</sup>
- h. Average heat flux in boiler (0-250 inches), 23,000 BTU/hr ft<sup>2</sup>

## II. SUMMARY

During the fifth quarter of the program, work proceeded on the topics abstracted below:

Significant effort was made in the past quarter in monitoring the fabrication of the advanced refractory alloys. Poor yields in the fabrication of the T-111 alloy have necessitated the melting of an additional ingot. The processing of this ingot is being expedited to minimize the delay to the program. All T-111 alloy material is now expected to be on hand by September 15, 1966.

All the drawings for the Alkali Metal Purification and Handling System for Corrosion Loop I (T-111) have been issued.

The sodium transfer system, used with the Prototype Corrosion Loop, is being modified for use in handling lithium for Corrosion Loop I. Fabrication of the lithium still is in progress.

### III. PROGRAM STATUS

#### A. MATERIALS PROCUREMENT

##### 1. T-111 Alloy - Fansteel Metallurgical Corporation

The processing of T-111 alloy tubing for Corrosion Loop I (T-111) is proceeding at Wolverine Tube Division of Calumet-Heccla. On May 4, 1966, the three remaining 2.93-inch OD x 9-inch long T-111 alloy tube hollows were extruded into base tubes each measuring approximately four-feet long.

In total eight T-111 alloy base tubes have been processed. Subsequently, the base tubes were cleaned, the ID honed, the OD polished, recleaned and inspected. A brief study was performed at General Electric to aid in selecting the annealing temperature to be used on the base tubes prior to tube reducing. Samples from the base tubes were annealed for one hour at 2500°, 2600°, 2700°, 2800°, 2900°, and 3000°F in vacuum. The samples were examined by metallographic and microhardness techniques.

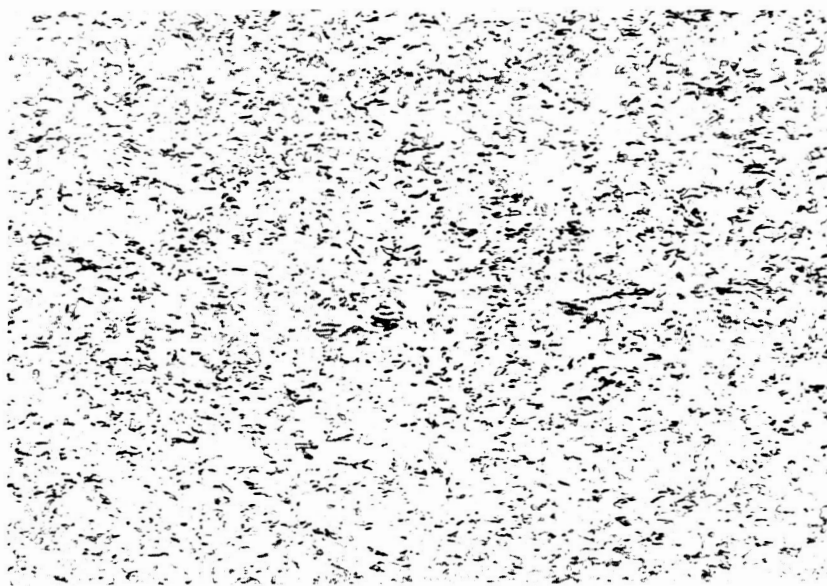
As can be seen in Figures 1 through 4, recrystallization has occurred in all of the annealed specimens. The microhardness and grain size data is presented in Table I. A one hour at 2700°F anneal was selected to obtain a desirable combination of hardness and grain size. Subsequently, the eight base tubes were wrapped in tantalum foil and annealed for one hour at 2700°F at a pressure of less than  $1 \times 10^{-5}$  torr by Wolverine Tube.

On July 12, six of these base tubes were successfully given a 50 percent reduction by Wolverine Tube to an intermediate tube size.

On May 9-11, 1966, the conditioned 4.25-inch diameter billets from the fourth T-111 ingots (No. 111-D-1102) were rolled to 3.5-, 3.25-, 2.75-, and 2.25-inch diameters at 1700°F in air at Braeburn Alloy Steel.

Electron beam melting of a fifth ingot, which was necessary when a poor yield was obtained from the fourth ingot, was completed at Fansteel on May 9, 1966. The fifth EB melted Ta-8W alloy ingot was forged into 4.0-inch diameter electrode stock on May 16, 1966, at Anderson-Schumaker, Chicago,





a) As Extruded



b) Annealed, 2500°F - 1 hour

C2004-1

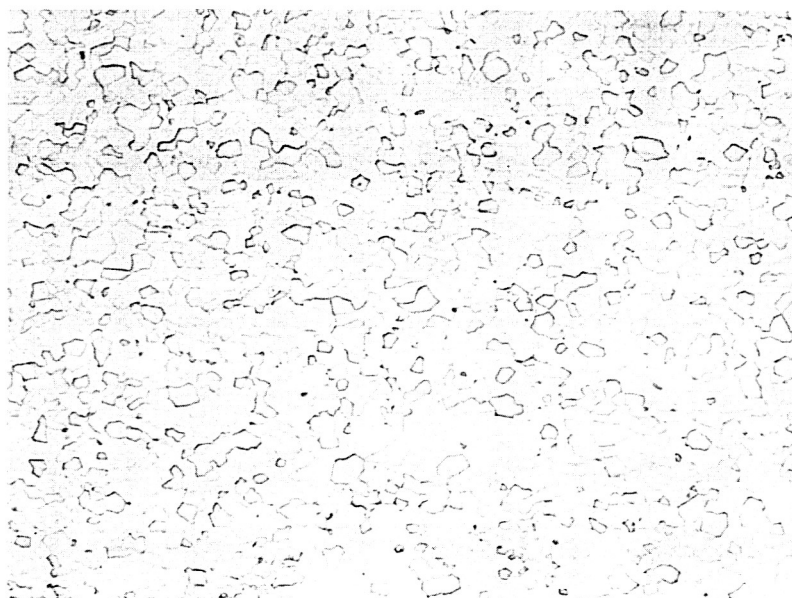
Figure 1. Transverse Microstructure of T-111 Alloy Extruded Base Tube.

Etchant: 30gmNH<sub>4</sub>F-20mlH<sub>2</sub>O-50mlHNO<sub>3</sub> Mag.: 100X

a) B830114 b) B830211



a) Annealed, 2600°F - 1 hour



b) Annealed, 2700°F - 1 hour

C2004-2

Figure 2. Transverse Microstructure of T-111 Alloy Extruded Base Tube.

Etchant: 30gmNH<sub>4</sub>F-20mlH<sub>2</sub>O-50mlHNO<sub>3</sub> Mag.: 100X

a) B830311 b) B830411



a) Annealed, 2800°F - 1 hour



b) Annealed, 2900°F - 1 hour

Figure 3. Transverse Microstructure of T-111 Alloy Extruded Base Tube.

Etchant: 30gmNH<sub>4</sub>F-20mlH<sub>2</sub>O-50mlHNO<sub>3</sub> Mag.: 100X

a) B830511 b) B830611

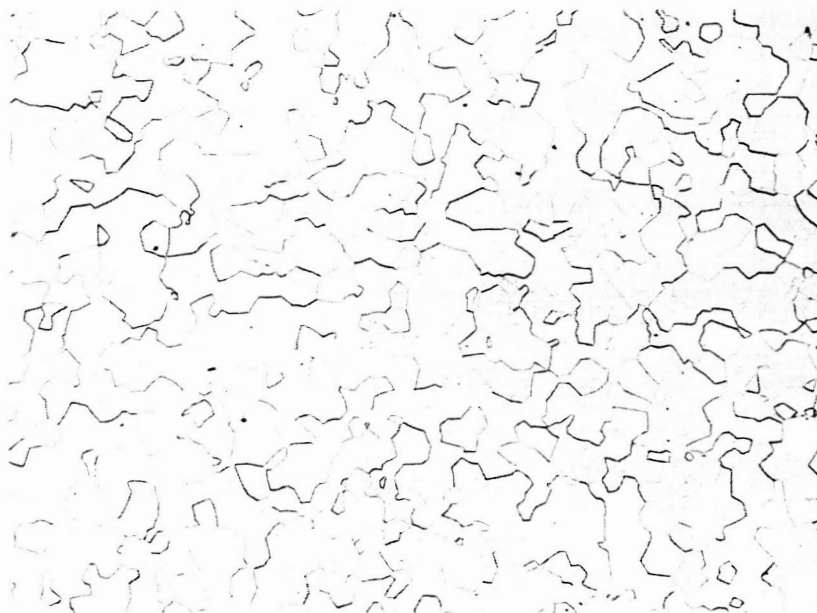


Figure 4. Transverse Microstructure of T-111 Alloy Extruded Base Tube After Annealing at 3000°F for One Hour in Vacuum.

Etchant: 30gmNH<sub>4</sub>F-20mlH<sub>2</sub>O-50mlHNO<sub>3</sub> Mag.: 100X  
(B830711)

TABLE I. MICROHARDNESS AND GRAIN SIZE OF UNANNEALED AND ANNEALED<sup>(a)</sup>  
T-111 ALLOY HEAT NUMBER 111-D-1670 EXTRUDED BASE TUBE

Annealing Temperature °F	Microhardness <sup>(b)</sup> DPH	ASTM Grain Size <sup>(c)</sup>
As Extruded	314	(d)
2500	217	7.5
2600	212	7.5
2700	210	7.5
2800	216	7.0
2900	214	6.5
3000	217	6.0

(a) Specimens annealed for one hour at pressures less than  $1 \times 10^{-5}$  torr.

(b) Average of four impressions at mid-wall location; 450-gram load.

(c) Intercept (or Heyn) procedure used.

(d) No value, specimen heavily cold worked.

Illinois, and subsequently arc melted to the T-111 composition on May 25, 1966, at Fansteel. The resulting 7.5-inch diameter arc melted ingot was machined to 6.72 inches, sectioned into two equal lengths, and shipped to Canton Drop Forging Company for extrusion. The two billets were canned in a molybdenum lined Type 304 stainless steel seamless pipe by heliarc welding and extruded on June 14, 1966. The extrusion parameters are presented in Table II. After extrusion, the cans were removed and the two billets were conditioned to 4-inch diameter and subsequently rolled to approximately 3-inch diameter on July 1, 1966. The rolling was performed at 800°F in air at Braeburn Alloy Steel.

It is presently expected that all of the T-111 alloy material required for loop construction will be on hand by September 15, 1966.

## 2. Mo-TZC Alloy - Climax Molybdenum Company

The 2-inch diameter rod was shipped on April 18, 1966. The remaining 2.125-inch diameter rod was rolled at 2400°F to 1.3-inch diameter on April 21, 1966, by Climax. The rod was subsequently machined to 1-inch diameter and shipped on May 4, 1966. The chemical analyses of the ingot and stress-relieved rod, as supplied by Climax, are shown in Table III. The mechanical properties, also supplied by Climax, are presented in Table IV.

## 3. Cb-132M Alloy - Universal Cyclops Steel Corporation

The remelted 5.375-inch diameter Cb-132M alloy ingot was canned in molybdenum and was extruded to 3.75-inch diameter at DuPont on April 25, 1966. The extrusion parameters are presented in Table V. The extruded bar was conditioned to 3.375-inch diameter and sectioned into two lengths. One billet length was stress-relieved at 2300°F for one hour in vacuum, machined, recanned in molybdenum, and extruded at 2400°F to approximately 2.25-inch diameter. This billet currently is being tested and inspected, and will be used to produce 2.0-inch diameter rod. The second billet length, which will be used to produce 1.0-inch diameter rod, was given a recrystallization anneal at 3200°F for one hour in vacuum, machined, recanned in molybdenum, and extruded at 2900°F to approximately 2.25-inch diameter. The

TABLE II. EXTRUSION PARAMETERS FOR THE 6.72-INCH  
DIAMETER MACHINED T-111 ALLOY INGOT (111-D-1765)\*

---

Machined Ingot Size - 6.72-inch diameter

Can Size - 7-1/4-inch OD x 1/4-inch thick wall Type 304 SS seamless pipe  
with a molybdenum foil liner, a 2-inch thick Type 304 SS nose  
block and a 1/2-inch thick Type 304 SS back-up block contained  
within the can.

Leader Block/Follow-up Block - 7-1/16-inch diameter x 6-inch long mild  
steel

Container Size - 7.51-inch ID

Die Size/Design - 4.5-inch ID/conical

Die Coating - None

Extrusion Ratio - 2.7/1

Lubricant - Hot die grease similar to Fiske 604

Furnace Temperature/Soak Time in Salt Bath - 2200°F/2 hours

Extrusion Pressure (Hydraulic) - 1,800 psi peak  
1,500 psi runout

Maximum Allowable Pressure - 3,850 psi

Cooling Procedure - Air cooled

Extrusion Direction of Ingot - Top of ingot was extruded through die  
first with hot top removed.

---

\* Extruded at Canton Drop Forging Company, Canton, Ohio, on June 14,  
1966.

TABLE III. CHEMICAL ANALYSES OF Mo-TZC ALLOY (HEAT NO. TZC-4431) PRODUCED BY CLIMAX MOLYBDENUM COMPANY

<u>Material</u>	<u>Sample Location</u>	<u>Chemical Analyses</u>					
		<u>%</u>			<u>ppm</u>		
		<u>C(a)</u>	<u>Ti(b)</u>	<u>Zr(b)</u>	<u>O(c)</u>	<u>H(c)</u>	<u>N(c)</u>
Ingot	Top - center	0.13	1.21	0.18			
	Top - mid-radius	0.13	1.20	0.14			
	Top - edge	0.13	1.21	0.21			
	Bottom - center	0.13	1.19	0.15			
2-inch diameter rod	Cross section	0.13			4	<1	2
1-inch diameter rod	Cross section	0.13			4	<1	<1

- (a) Combustion analysis.
- (b) X-ray fluorescence analysis.
- (c) Vacuum fusion analysis.



TABLE IV. MECHANICAL PROPERTIES(a) OF Mo-TZC ALLOY  
(HEAT NO. TZC-441) PRODUCED BY CLIMAX MOLYBDENUM COMPANY

Specimen Location	Room Temperature Tensile Properties (b)				Stress-Rupture Life at 2400°F and 30,000 psi in Vacuum of 3 x 10 <sup>-5</sup> Torr (c)			
	Ultimate Strength 1,000 psi	0.2% Yield		Elong. %	Life Hours	Creep Rate %/Hour	Elong. %	Reduction in Area %
		Strength 1,000 psi	Strength 1,000 psi					
1-inch diameter bar, mid-radius	118.4	106.0	106.0	15.5	35.5	0.052	28	86.1
	118.0	109.1	109.1	32.5	30.2	0.043	30	86.4
2-inch diameter bar, mid-radius	104.1	96.1	96.1	1.5	73.2	0.03	33	64
	106.1	97.1	97.1	2.0	101.9	0.03	29	79

- (a) Tensile and stress-rupture specimens were machined from Mo-TZC alloy bar following annealing for one hour at 2400°F. The axes of the specimens were parallel to the rolling direction. The specimens had a 0.250-inch diameter with a 1.250-inch gauge length and were hand polished prior to testing.
- (b) Tensile properties were determined using a strain rate of 0.005 inch/inch/min up to 0.6% offset and then 0.050 inch/inch/min to fracture.
- (c) Stress-rupture specimens were held for a half-hour at 2400°F before load was applied.

TABLE V. EXTRUSION PARAMETERS FOR THE 5.36-INCH  
DIAMETER MACHINED Cb-132M ALLOY INGOT(1)

---

---

Machined Ingot Size	-	5.36-inch diameter x 10.16 inch long
Can Size	-	5.95-inch diameter molybdenum
Leader Block	-	Steel block heated to 2000°F
Follow-up Block	-	Two one-inch thick carbon discs heated to 2000°F followed by one carbon disc at room temperature.
Container Size	-	6-inch ID
Die Size/Design	-	3.75-inch ID/conical
Die Coating	-	ZrO <sub>2</sub>
Extrusion Ratio	-	2.56/1
Lubricant	-	Glass
Ingot Temperature <sup>(b)</sup>	-	3120°F
Extrusion Pressure	-	76,000 psi peak on stem
Extrusion Press Size	-	2,750 tons
Cooling Procedure	-	Air

---

---

(a) Extruded at DuPont on April 25, 1966.

(b) Heated by induction in argon.

extrusion parameters for both billets are presented in Table VI. The latter two extrusions were performed by Nuclear Metals, West Concord, Massachusetts, on June 3, 1966. The second billet length was then stress-relieved at 2300°F for one hour in vacuum, machined, recanned in molybdenum, and extruded by Nuclear Metals on July 15, 1966, at 2400°F through a 1.75-inch diameter die. The extrusion parameters are presented in Table VII. Decanning is now in progress.

#### B. ALKALI METAL PURIFICATION AND HANDLING

The bakeout oven for the high-vacuum system on the lithium dolly was received, insulated, and wired for use. All the drawings for the Alkali Metal Purification and Handling System for Corrosion Loop I (T-111) have been issued.

The sodium and potassium transfer system used for the Prototype Corrosion Loop<sup>(1)</sup> was inspected to determine the extent of the modification required to prepare it for use in loading and sampling the lithium and potassium for Corrosion Loop I (T-111). The only required modification of the potassium system will be the installation of a level probe well in the charge pot. Modifications of the sodium transfer system to handle lithium will be more extensive. All components which were in contact with sodium will be scrupulously cleaned or replaced with new components prior to re-assembly, with the exception of the disposal tank. The disposal tank is merely a container for collecting used alkali metal for subsequent disposal. Sodium from the Prototype Loop Test will be drained from this tank prior to re-use.

The analysis of the metallic impurities in the 29-pound batch of hot trapped lithium was received and is shown in Table VIII. This material was also analyzed for oxygen by General Atomic, San Diego, California, using the fast neutron activation technique. The results were  $106 \pm 16$  and  $123 \pm 13$  ppm oxygen. The nitrogen concentration, reported previously<sup>(2)</sup>, was  $< 10$  ppm.

Fabrication of the lithium still is in progress. The lithium still receiver shell has been rolled and welded. Necessary stainless steel components for the still pot and condenser are being machined. The Cb-1Zr

TABLE VI. EXTRUSION PARAMETERS FOR THE 3.75-INCH DIAMETER  
MACHINED Cb-132M ALLOY BILLETS(a)

---

Machined Billets Sizes - Billet No. 1<sup>(b)</sup> - 3.75-inch diameter x 12-inch long  
Billet No. 2<sup>(c)</sup> - 3.75-inch diameter x 10-inch long

Can Size - 3.95-inch diameter molybdenum

Leader Block - Mild steel heated to 1500°F

Container Size - 4.05-inch ID

Die Size/Design - 2.625-inch ID/conical

Die Coating -  $ZrO_2$

Extrusion Ratio - 2.4/1

Lubricant - Glass

Billet Temperature<sup>(d)</sup> - Billet No. 1 - 2400°F  
Billet No. 2 - 2900°F

Extrusion Pressure - Billet No. 1 - 750-690 tons runout  
Billet No. 2 - 510-370 tons runout

Extrusion Press Size - 1,400 tons

Cooling Procedure - Air

---

- (a) Extruded at Nuclear Metals, West Concord, Mass., on June 3, 1966.
- (b) Billet No. 1 will be used to make the 2-inch diameter bar.
- (c) Billet No. 2 will be used to make the 1-inch diameter bar.
- (d) Heated by induction in argon.

TABLE VII. EXTRUSION PARAMETERS FOR THE 2.0-INCH  
DIAMETER MACHINED Cb-132M ALLOY BILLET<sup>(a)</sup>

---

Machined Billet Size - 2.0-inch diameter x 12-inch long

Can Size - 3.95-inch diameter molybdenum

Leader Block - None

Follow-up Block - Mild steel heated to 900°F

Container Size - 4.05-inch ID

Die Size/Design - 1.75 ID/conical

Die Coating -  $ZrO_2$

Extrusion Ratio - 3.0/1

Lubricant - Glass

Billet Temperature<sup>(b)</sup> - 2400°F

Extrusion Pressure - 700 tons

Extrusion Press Size - 1,400 tons

Cooling Procedure - Air

---

(a) Extruded at Nuclear Metals, West Concord, Mass., on  
July 15, 1966.

(b) Induction heated in argon.

TABLE VIII. SPECTROGRAPHIC ANALYSIS OF LITHIUM AFTER  
HOT TRAPPING FOR 126 HOURS AT 1500°F

<u>Element</u>	<u>Concentration, ppm</u> <u>(in Lithium)</u>
Ag	<5
Al	5
B	<50
Be	<5
Ca	5
Cb	<25
Co	<5
Cr	<5
Cu	28
Fe	5
K	<50
Mg	<5
Mn	<5
Mo	<5
Na	80
Ni	28
Pb	<25
Si	28
Sn	<25
Ti	<5
V	<25
Zr	<25

tubing for the level probe was received and accepted. Cb-1Zr tubing for the thermocouple well has not been received; however, its delivery should not delay fabrication of the still.

C. QUALITY ASSURANCE

All of the necessary specifications have been prepared and submitted to the NASA Program Manager for review. All of the Corrosion Loop I (T-111) design drawings have been approved by the NASA Program Manager.

#### IV. FUTURE PLANS

- A. Monitoring the fabrication of refractory alloy materials for Corrosion Loop I (T-111) will continue with special emphasis placed on meeting the new schedule.
- B. Fabrication of the critical loop components will be initiated upon receipt of the refractory alloy mill products.
- C. Fabrication of the Lithium Metal Purification and Handling System for Corrosion Loop I (T-111) will continue.



#### REFERENCES

- (1) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 7 for Period Ending April 15, 1965, NASA Contract NAS 3-2547, NASA-CR-54735, p 55.
- (2) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 4 for Period Ending April 15, 1966, NASA Contract NAS 3-6474.

PUBLISHED REPORTS

<u>Quarterly Progress</u>	<u>For Quarter Ending</u>
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Report No. 3 (NASA-CR-54911)	January 15, 1966
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